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OCT 10 2002

TC 1700

WITCO CORPORATION
OSI SPECIALTIES GROUP
TARRYTOWN, NEW YORK

8

SUBJECT

Scale Up of TMS & NEMALA w/ M*M*

DATE

TEST NO. 3196-B

1 Obj: To scale up reaction & produce TT Samples

2
3 Materials: NEMALA 150g (1.51 moles)
4 TMS 220g (1.80 moles)
5 M*M* 0.25 mL (34 ppm)6
7 Equipment: 500 mL 3 neck flask equipped w/ magnetic stirrer,
8 addition funnel & dry ice condenser w/ N₂ inlet.9
10 Procedure: Charge TMS to flask & stir. Charge M*M* & heat
11 to 60°. Charge NEMALA thru addition funnel & maintain
12 temp < 80°C. Hold @ 90° for 1 hr. & heat to 105°.
13 GC analysis & strip conditions in Comment section14
15 Time Temp Comments

16 8:35 Heating TMS to 60° Charged M*M*.

17 8:51 67.5 Charging NEMALA w/ addition funnel about 1 drop/sec.

18 9:02 62.9 Increased feed rate to about 3 drops/sec.

19 * Started adding at 67.5 w/ mantle set on 60°C. Temp dropped
20 to 62.5 before starting to climb. *

21 9:07 64.5

22 9:08 Can see some bubbles forming in flask

23 9:12 65.3

24 9:20 64

25 9:30 60.4 Increased mantle temp to 90°. Hold @ 90° until 10:30

26 9:37 80.1 Addition complete. Still lots of bubbles in flask.

27 - or reflux? of TMS (Temp now 92.1) 9:40

28 9:50 91.8

29 10:00 89.8

30 10:30 89.9 Increased temp to 105°.

31 10:50 103.8

32 11:50 105.7

33 12:30 105.1

DATA BY

Beth A. Gaylor

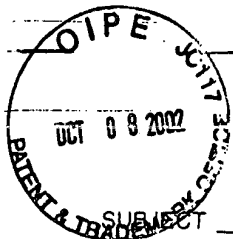
DATE

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Cathy L. Clegg

DATE

CROSS REFERENCES



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9

Cont'd PD-3196-8

DATE

TEST NO. 3196-9

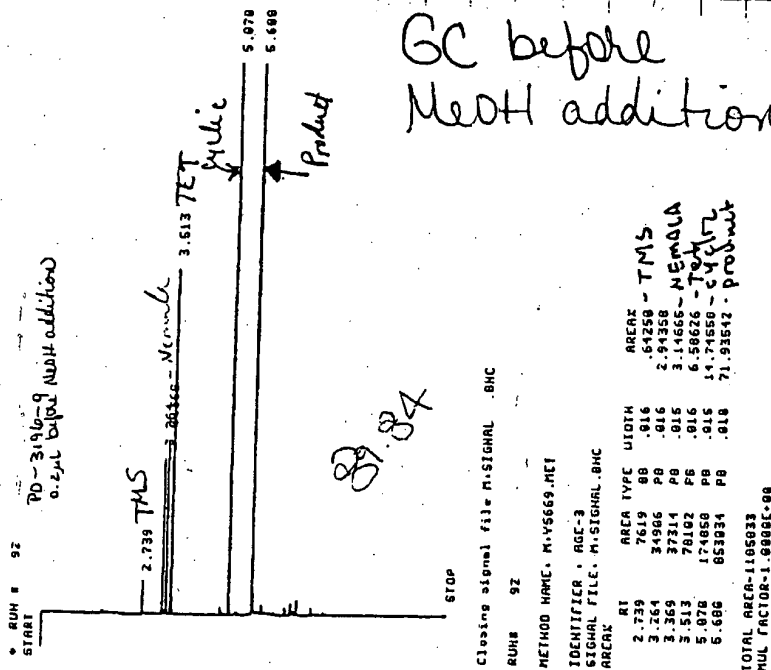
Time	Notes
2:00	105.0
2:30	104.3
3:00	Cooling to Run GC
3:40	Charging 65ml MeOH thru addition funnel to condenser cyclic.
3:49	Temp @ 76.5 on ice. Heavy foaming w/ addition of MeOH.
3:50	All MeOH added.

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GC before
MeOH addition



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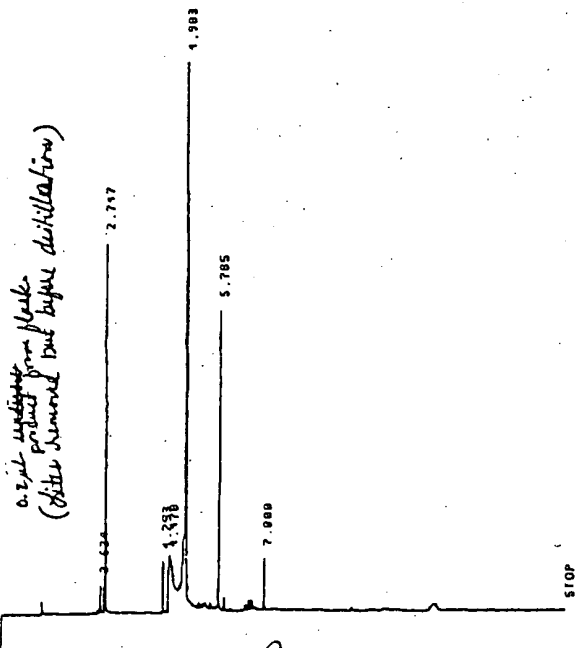
Original GC in Analytical Results Folder

TEST NO. 3196-10

Pat	Head
-----	------

1	Time	Temp	Comments
2	8:30		Sampled for GC analysis. Set up to Strip Lites.
3	9:23		Vacuum @ 51 mm Hg. NO heat. Removing Lites.
4	9:33	22.6	Heating to 60°C. Problems w/ bumping.
5	9:42		Shut down vacuum & GC'd Lites. 49% MeOH.
6			Bumping resulted in 20% product.
7	9:57		Stripping w/o vacuum.
8	10:30	113/71	Lites coming over.
9	10:40		Product building color & head temp remains ~ 70.
10			Shutting down to run GC.
11	11:02	25	Vacuum @ 24 mm Hg. Using slight N ₂ spurge.
12	11:11	36/27	" " " " Lites coming over.
13	11:21	50/33	Increased temp to 70°C.
14	11:36	64/40	Very little Lites (if any) coming over.
15	11:45	74/43	Shutting down for Salaries Review.
16			

GC of flask



Closing signal file M.SIGNAL -BNC

RUN# 2003

ΠΕΡΙΛΗΨΗ

IDENTIFIER : GLC 3181-51-15-181E 375 : 831J11K301

SIGNAL FILE: N: SIGNAL .BNC

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2.624 7801 PB

4.293 12600 P8

1.478	90163	PV
4.883	7600030	10

50	57593	BB
51	582.8	BB

84 0600T 000-1

TOTAL AREA-7076154

00-10000-T-2017-2017

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[illegible]

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Age Group	1980	1990	2000	2010	2020
0-14	25	22	18	15	10
15-24	20	18	15	12	8
25-34	15	14	13	12	10
35-44	12	11	10	10	10
45-54	10	10	10	10	10
55-64	8	8	8	8	8
65-74	10	12	15	18	25
75+	5	5	5	5	5

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Abstract

100

Figure 1. Schematic representation of the experimental design. The subjects were divided into two groups: the control group and the experimental group. The control group received a standard diet and water, while the experimental group received a diet supplemented with 0.5% of the test substance. The subjects were divided into two groups: the control group and the experimental group. The control group received a standard diet and water, while the experimental group received a diet supplemented with 0.5% of the test substance.

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-DATA-BY

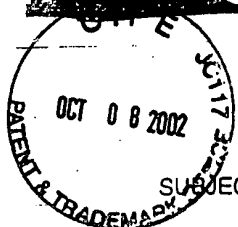
Both (A & B) all

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Cathy L. Clegg

DATE:



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OSI SPECIALTIES GROUP
TARRYTOWN, NEW YORK

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TC 1700

DATE

SUBJECT Could PD-3196-8

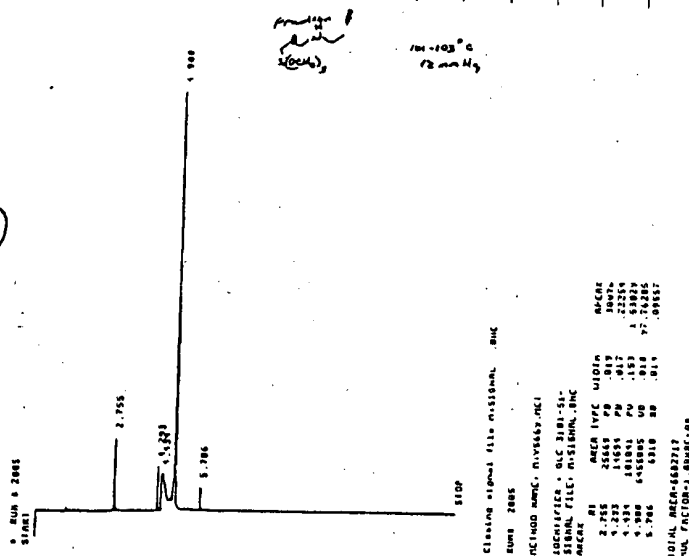
TEST NO. 3196-11

Time	Temp	Comments
1:05	25	Vacuum @ 23 mm Hg. Heating to 90°C
1:23		Broke vacuum to remove N ₂ inlet. Vacuum ~ 50.
1:27	83/37	Vacuum @ 10 mm Hg.
1:31	85/33	No more lites. Broke vacuum & removed Receiver.
		Replaced w/ 250 mL receiver. Put on the dry ice/ice.
1:35	90/31	Heating to 120°C. Vacuum @ 9 mm Hg. No sparge.
1:45	102/29	Starting to bubble. " " 14 mm Hg
1:53	104/100	Distilling rapidly. Insulated flask. Vacuum @ 12 mm Hg
2:03	103/101	" " Vacuum @ 11 mm Hg.
2:24	106/94	Vacuum @ 14 mm Hg.
2:31	105/103	" " 12 mm Hg Distilling over
2:48	98/96	" " 9 mm Hg
3:06	108-3	" " 9 mm. Broke vacuum. Cooling.

Cut 1 = 186.43 g

Cut 2 = 86.79 g = 273.22 g total grams

Fraction
#1
of
Distillation



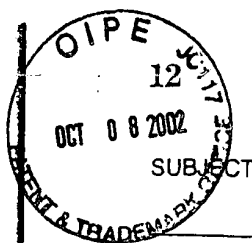
82% yield
isolated

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DATE

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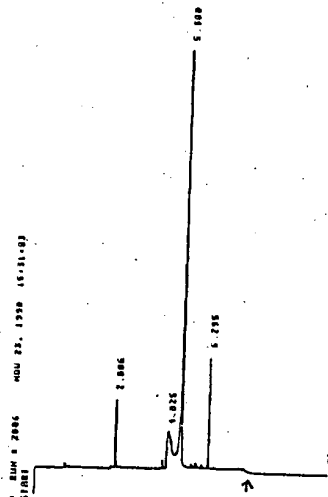
TC 1700

TEST NO. 3196-12

Cont'd PD-3196-8

There was
a front inlet
pressure shutdown
on Nov 22. 1999
Think it's ok
go ahead
and run
w/ front inlet

2nd
inlet
with
silicon

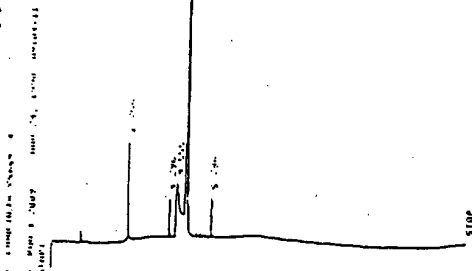


Closing signal file: n1signal.dmc
RUN: 2006 7 NOV 23, 1999 15:11:07
METHOD NAME: N1SIGNAL.DMC
IDENTIFIED: GLC 3101-S1-
SIGNAL FILE: n1signal.dmc
AREAS
RT AREA TYPE WIDTH
2.086 20861 PG 823 15029
5.035 20861 PG 823 15029
5.189 20861 PG 823 15029
TOTAL AREA: 725178
NUM FACTOR: 1.000E+00

Distillation Cut #2

Mixture of Fractions
1 & 2

Plot of 1st Distillation



Closing signal file: n1signal.dmc
RUN: 2009
METHOD NAME: N1SIGNAL.DMC
IDENTIFIED: GLC 3101-S1-
SIGNAL FILE: n1signal.dmc
AREAS
RT AREA TYPE WIDTH
2.755 33611 PG 819 41064
3.361 33611 PG 819 41064
4.064 33611 PG 819 41064
4.426 33611 PG 819 41064
4.884 33611 PG 819 41064
5.189 33611 PG 819 41064
5.786 33611 PG 819 41064
TOTAL AREA: 725178
NUM FACTOR: 1.000E+00

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Beth C. Gaylor

DATE

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DATE

CROSS REFERENCES

Original GC Analysis Results Folder

OCT 08 2002

WITCO CORPORATION
OSI SPECIALTIES GROUP
TARRYTOWN, NEW YORK

SUBJECT

Scale Up of γ -1205 & Nemala
(γ -15230)

DATE

TEST NO. PD-3219-5A

1 Obs. large scale hydroxylation of γ -1205 & N-ethylmethallyl-
2 amine by using M* M*. Sample to be sent to Tarrytown
3 for evaluation.
4

5 Materials: γ -1205 381.3 g (5% excess after 97% purity)
6 Nemala 260g
7 M* M* 0.65 mL (50 ppm)
8

9 Equipment: 4 neck ~~fl~~ flask equipped w/ N₂ blowby, addition
10 funnel, distillation apparatus w/ cold finger, thermocouple &
11 magnetic stirrer.
12

13 Procedure: Heat γ -1205 & M* M* to 90°C & drip in Nemala.
14 Heat to 110°C & hold 1 hr.
15

16 Time Temp Comments

17 1:00 Heating to 90°C.

18 1:30 99.7 Dipping in Nemala.

19 1:20 101.9 "

20 1:30 101.6 "

21 2:12 112.9 "

22 2:32 103.0

23 2:42 98.6 All Nemala added. Heating to 110°C. Hold until 3:30

24 3:00 119.8

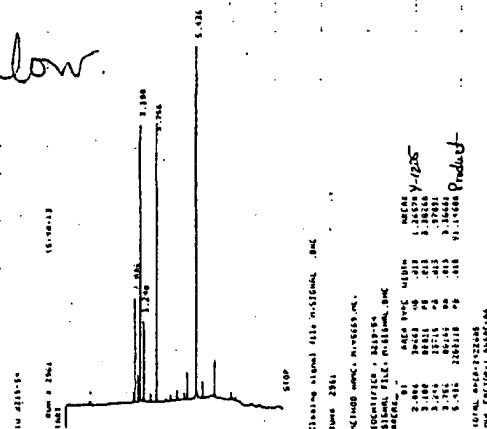
25 3:30 Cooling to take HPLC. Shown Below.

26 By GC, Nemala RT is 3.101

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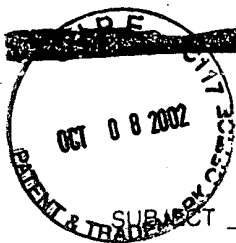
Beth A. Kapp

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DATE



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OSI SPECIALTIES GROUP
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55

Cont'd PD-3219-54

DATE

TEST NO PD-3219-55

Time	Temp	Comments (Lites Strip)	
8:55		Vacuum @ 26 mm Hg. Heating to 50°C.	
9:12	41.9	Head temp = 25°C. A few drops coming over.	
9:18	63.6	HT = 28°C	
9:37	84.1	HT = 34°C. Very few drops coming over.	
9:45	94.5	HT = 37°C. Lites are @ 1 drop per 7 seconds.	
10:14	98.5	HT = 36°C. Lites are @ 1 drop per 14 seconds.	
10:20	106.4	HT = 50°C. Lites are @ 1 drop per 5 seconds.	
10:35	116.0	HT = 57°C. " " " " " "	
10:54	122.4	HT = 60°C. " " " " " 3 seconds. Vacuum @ 30 mm Hg.	
11:13	126.4	HT = 68°C.	
11:47	128.5	HT = 64°C. Cooling to 100°C over lunch.	
11:55			
12:40		Heating to 150°C.	
1:12		Lowered vacuum & removed lites. Temp ~ 132°C. Pulling vacuum & heat lites GC. (27 mm Hg)	
1:19	130.8	HT = 80°C.	
1:38	129.8	HT = 90°C.	
2:15	130.1	HT = 90°C.	
2:30	130.2	HT = 88°C.	
4:00	130.6	HT = 88°C. vacuum. 27 mm Hg. Shut down distillation placed N ₂ blowby on apparatus. S.C.	

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DATE

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OCT 08 2002

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TARRYTOWN, NEW YORK

SUBJECT CONFID PD-3219-SA

DATE

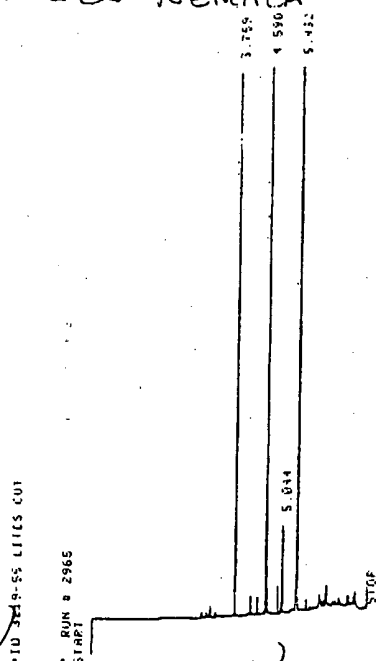
TEST NO. PD-3219-Sb

1 Time Temp Comments
 2 8:15 Emptied receiver - collected 150.43 g. Product GC'd.
 3 8:20 Vacuum @ 30 mm Hg. Heating to 150°C.
 4 8:48 132.8 Boiling HT = 72°. Beginning to distill.
 5 9:05 129.8 HT = 102°C. Distilling @ 1 drop per 2 seconds.
 6 9:30 129.8 HT = 98°C. Wrapped w/ foil. Distilling rapidly.
 7 HT = 118°C. Product went to NUS ???
 8 10:01 134.4 HT = 127°C.
 9 10:05 Turned off heat. Very little left in flask.
 10 10:45 GC of receiver. 100% Product: same as cut #1.
 11 Collected 328.07 g in second cut.

Total Product distilled = 484.55 g

$$\frac{484.55 \text{ g}}{231} = 2.098 \text{ moles}$$

$$\frac{2.098 \times 15230}{2.626 \text{ NEMAL}} \times 100 = 79.89\% \text{ Isolated yield.}$$



Closing signal file M: SIGNAL BIN

RUN# 2965

METHOD NAME: M: 5669.MET

IDENTIFIER: 3219-55 LITE

SIGNAL FILE: M: SIGNAL.BIN

APCAR:

PI	APCAR	TIME	HEIGHT	AREA
3.759	105965	BB	.014	5.01197 4.1205 Impurity
4.590	121087	BB	.014	6.48251 Cyclic ??
5.044	10196	BB	.015	.00279
5.432	103310	BB	.017	97.00973 Product

TOTAL APCAR=1877158
MUL FACTOR=1.00000000

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Beth A. Kasper
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PATENT & TRADEMARK OFFICE
OCT 08 2002

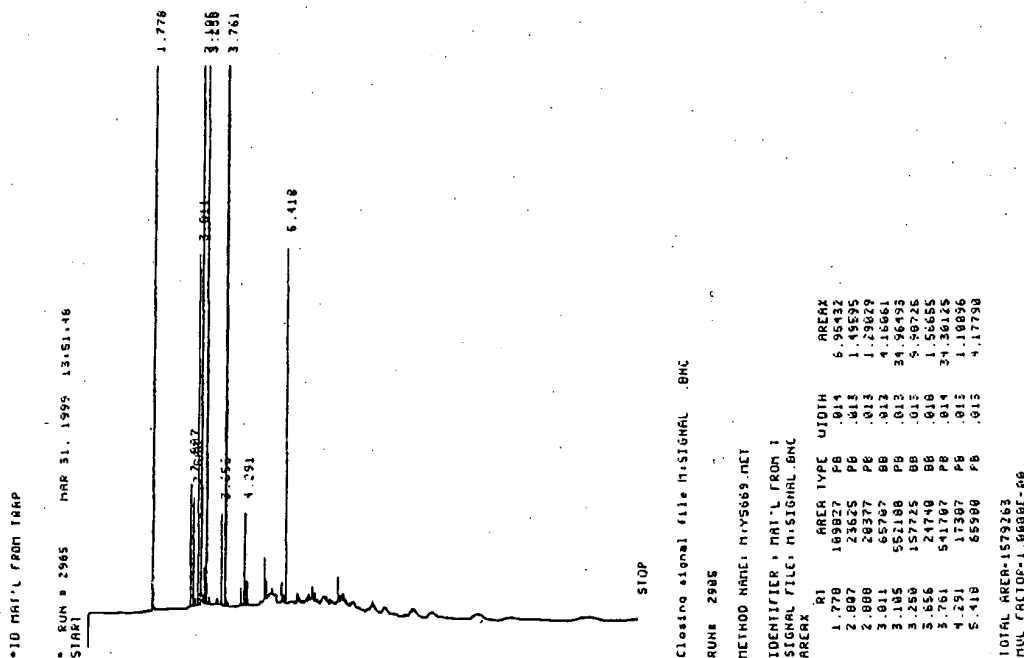
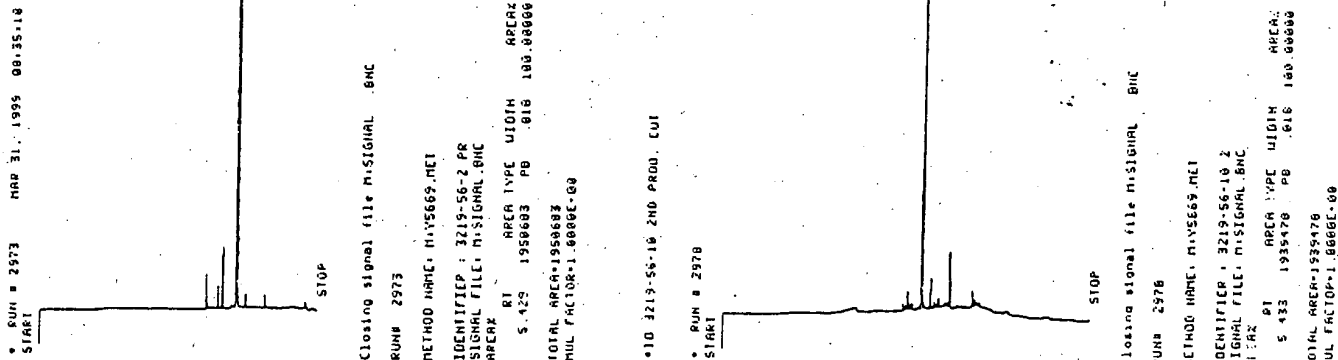
WITCO CORPORATION
OSI SPECIALTIES GROUP
TARRYTOWN, NEW YORK

57

SUBJECT: WITCO PO-3219-54

DAT.

TEST NO. PO-3219-57



DATA BY

Beth A. Rogers

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